DRAFT

FLUOROMONOMERS, IXM AND PPA MANUFACTURING PROCESSES EMISSIONS TEST PROTOCOL

THE CHEMOURS COMPANY FAYETTEVILLE, NORTH CAROLINA

Prepared for:

THE CHEMOURS COMPANY

22828 NC Hwy 87 W Fayetteville, North Carolina 28306

Prepared by:

WESTON SOLUTIONS, INC.

1400 Weston Way P.O. Box 2653 West Chester, Pennsylvania 19380

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1. INTRODUCTION

1.1 FACILITY AND BACKGROUND INFORMATION

The Chemours Fayetteville Works is located in Bladen County, North Carolina, approximately ten miles south of the city of Fayetteville. Chemours operating areas on the site include the Fluoromonomers, IXM and Polymer Processing Aid (PPA) manufacturing areas, Wastewater Treatment, and Powerhouse.

1.2 TEST OBJECTIVES

The specific objectives for this test program are as follows:

- Measure the emissions concentrations and mass rates of HFPO Dimer Acid from the Division stack, VE South Scrubber stack and PPA stack which are located in the Fluoromonomers, Nafion® and PPA processes.
- Monitor and record process and emissions control data in conjunction with the test program.
- Provide representative emissions data.

1.3 TEST PROGRAM OVERVIEW

During the emissions test program, the concentrations and mass rates of HFPO Dimer Acid will be measured on the three sources (Division, VE South Scrubber and PPA Stacks).

Tables 1-1, 1-2 and 1-3 provide a summary of the test locations and the parameters to be measured along with the sampling/analytical procedures to be followed.

Following this Introduction, a description of the processes is provided in Section 2. Section 3 provides a description of the test locations. The sampling and analytical procedures are provided in Section 4. Test procedures are described in Section 5. An outline of the Emissions Test Report is provided in Section 6 and Quality Assurance procedures are provided in Section 7.

Table 1-1
Sampling Plan for Division Stack Gas

| Sampling Point & Location | Division Stack | | | | |
|---|-----------------------------|---|-------------------|--------|---|
| Number of Tests: | 3 | | | | |
| Parameters To Be Tested: | HFPO Dimer Acid | Volumetric Flow Rate and Gas Velocity | Carbon Dioxide | Oxygen | Water Content |
| Sampling or Monitoring Method | EPA M0010 | EPA M1, M2, M3A, and M4 in conjunction with M0010 tests | EPA M3A | | EPA M4 in conjunction with M0010 tests |
| Sample Extraction/ Analysis Method(s): | LC/MS/MS | NA ⁶ | NA | | NA |
| Sample Size | $> 3m^3$ | NA | NA | NA | NA |
| Total Number of Samples Collected ¹ | 3 | 3 | 3 | 3 | 3 |
| Reagent Blanks (Solvents, Resins) ¹ | 1 set | 0 | 0 | 0 | 0 |
| Field Blank Trains ^l | 1 per run | 0 | 0 | 0 | 0 |
| Proof Blanks ¹ | 1 per train | 0 | 0 | 0 | 0 |
| Trip Blanks ^{1,2} | 1 set | 0 | 0 | 0 | |
| Lab Blanks | 1 per fraction ³ | 0 | 0 | 0 | 0 |
| Laboratory or Batch Control Spike Samples (LCS) | 1 per fraction ³ | 0 | 0 | 0 | 0 |
| Laboratory or Batch Control Spike Sample Duplicate (LCSD) | 1 per fraction ³ | 0 | 0 | 0 | 0 |
| Media Blanks | 1 set ⁴ | 0 | 0 | 0 | 0 |
| Isotope Dilution Internal Standard Spikes | Each sample | 0 | 0 | 0 | 0 |
| Total No. of Samples | 95 | 3 | 3 | 3 | 3 |

Key:

¹ Sample collected in field.

² Trip blanks include one XAD-2 resin module and one methanol sample per sample shipment.

³ Lab blank and LCS/LCSD includes one set per analytical fraction (front half, back half and condensate).

⁴ One set of media blank archived at laboratory at media preparation.

⁵ Actual number of samples collected in field.

⁶ Not applicable.

Table 1-2 Sampling Plan for VE South Scrubber Stack

| Sampling Point & Location | VE South Scrubber Stack | | | | |
|--|-----------------------------|---|-------------------|--------|---|
| Number of Tests: | 3 | | | | |
| Parameters To Be Tested: | HFPO Dimer Acid | Volumetric Flow Rate and Gas Velocity | Carbon Dioxide | Oxygen | Water Content |
| Sampling or Monitoring Method | EPA M0010 | EPA M1, M2, M3A, and M4 in conjunction with M0010 tests | EPA M3A | | EPA M4 in conjunction with M0010 tests |
| Sample Extraction/ Analysis Method(s): | LC/MS/MS | NA ⁶ | NA | | NA |
| Sample Size | > 3m ³ | NA | NA | NA | NA |
| Total Number of Samples Collected ¹ | 3 | 3 | 3 | 3 | 3 |
| Reagent Blanks (Solvents, Resins) ¹ | 1 set | 0 | 0 | 0 | 0 |
| Field Blank Trains ¹ | 1 per run | 0 | 0 | 0 | 0 |
| Proof Blanks ¹ | 1 per train | 0 | 0 | 0 | 0 |
| Trip Blanks ^{1,2} | 1 set | 0 | 0 | 0 | |
| Lab Blanks | 1 per fraction ³ | 0 | 0 | 0 | 0 |
| Laboratory or Batch Control Spike Samples (LCS) | 1 per fraction ³ | 0 | 0 | 0 | 0 |
| Laboratory or Batch Control Spike Sample Duplicate (LCSD) | 1 per fraction ³ | 0 | 0 | 0 | 0 |
| Media Blanks | 1 set ⁴ | 0 | 0 | 0 | 0 |
| Isotope Dilution Internal Standard Spikes | Each sample | 0 | 0 | 0 | 0 |
| Total No. of Samples | 95 | 3 | 3 | 3 | 3 |

Key:

¹ Sample collected in field.

² Trip blanks include one XAD-2 resin module and one methanol sample per sample shipment.

³ Lab blank and LCS/LCSD includes one set per analytical fraction (front half, back half and condensate).

⁴ One set of media blank archived at laboratory at media preparation.

⁵ Actual number of samples collected in field.

⁶ Not applicable.

Table 1-3
Sampling Plan for PPA Stack

| Sampling Point & Location PPA Stack | | | | kirineihelikääääkitäilienhuunnannannyanyanpanpanpan <u>yanyaaaaa</u> | |
|--|-----------------------------|---|-------------------|--|---|
| Number of Tests: | 3 | | | | |
| Parameters To Be Tested: | HFPO Dimer Acid | Volumetric Flow Rate and Gas Velocity | Carbon Dioxide | Oxygen | Water Content |
| Sampling or Monitoring Method | EPA M0010 | EPA M1, M2, M3A, and M4 in conjunction with M0010 tests | EPA M3A | | EPA M4 in conjunction with M0010 tests |
| Sample Extraction/ Analysis Method(s): | LC/MS/MS | NA ⁶ | NA | | NA |
| Sample Size | > 3m ³ | NA | NA | NA | NA |
| Total Number of Samples Collected ¹ | 3 | 3 | 3 | 3 | 3 |
| Reagent Blanks (Solvents, Resins)1 | 1 set | 0 | 0 | 0 | 0 |
| Field Blank Trains ¹ | l per run | 0 | 0 | 0 | 0 |
| Proof Blanks ¹ | 1 per train | 0 | 0 | 0 | 0 |
| Trip Blanks ^{1,2} | 1 set | 0 | 0 | 0 | |
| Lab Blanks | 1 per fraction ³ | 0 | 0 | 0 | 0 |
| Laboratory or Batch Control Spike Samples (LCS) | 1 per fraction ³ | 0 | 0 | 0 | 0 |
| Laboratory or Batch Control Spike Sample Duplicate (LCSD) | 1 per fraction ³ | 0 | 0 | 0 | 0 |
| Media Blanks | 1 set ⁴ | 0 | 0 | 0 | 0 |
| Isotope Dilution Internal Standard Spikes | Each sample | 0 | 0 | 0 | 0 |
| Total No. of Samples | 95 | 3 | 3 | 3 | 3 |

Key:

¹ Sample collected in field.

² Trip blanks include one XAD-2 resin module and one methanol sample per sample shipment.

³ Lab blank and LCS/LCSD includes one set per analytical fraction (front half, back half and condensate).

⁴ One set of media blank archived at laboratory at media preparation.

⁵ Actual number of samples collected in field.

⁶ Not applicable.

2. PROCESS DESCRIPTIONS

The Fluoromonomers, IXM and PPA areas are included in the scope of this test plan.

2.1 POLYMER PROCESSING AID (PPA) AREA

The PPA facility produces surfactants used to produce Chemours Teflon® as well as sales to outside producers of fluoropolymers.

Process streams are vented to a caustic wet scrubber (ACD-A1) and vented to a process stack (AEP-A1). The process inside of the building is under negative pressure and the building air is vented to the process stack (AEP-A1).

2.2 FLUOROMONOMERS & IXM AREAS

These facilities produce a family of fluorocarbon compounds used to produce Chemours products such as Nafion®, Krytox®, and Viton®, as well as sales to outside customers.

The following process streams are vented to the Division Waste Gas Scrubber (NCD-Hdr1):

- HFPO Refining
- VEN Crude Ether Process
- VEN Condensation
- Refined VE Process

The Division Waste Gas Scrubber is vented to a process stack (NEP-Hdr1). In addition, the following building air systems are vented to this stack:

- HFPO RV Catch Pots
- HFPO Tower Exhaust Blower
- VEN Permeators Catch Pot
- HDT Vent
- VEN Tower Exhaust Blower
- Analyzer Room Blower
- HFPO Crude Dryer Moisture Analyzer Vent

The following processes are vented to the VE South Waste Gas Scrubber (NCD-Hdr2):

Refining

- Condensation
- Agitated Bed Reactor

The VE South Waste Gas Scrubber is vented to a process stack (NEP-Hdr2). In addition, the following building air systems are vented to this stack:

- Permeators
- RV Catch Pots
- Tower HVAC
- Nitrogen Supply to Catch Tanks
- Catalyst Feed Tank Pot Charge Vent

2.3 PROCESS PARAMETERS

During the test program, the following parameters will be monitored by Chemours and included in the emissions test report.

- PPA Process
 - o Caustic Wet Scrubber (ACD-A1)
 - Caustic recirculation flow rate
 - Differential pressure across the packing
- Fluoromonomers & IXM Processes
 - o Division Waste Gas Scrubber
 - Caustic recirculation flow rate
 - VE South Waste Gas Scrubber
 - Caustic recirculation flow rate

3. DESCRIPTION OF TEST LOCATIONS

3.1 DIVISION STACK

Two 6" ID test ports will be installed on the \sim 32" ID fiberglass stack. The ports will be placed \sim 30' (11.3 diameters) from the nearest downstream disturbance and 9' (3.4 diameters) from the stack exit. The four vents that enter the top of the stack and the one vent \sim 11' below are catch pots which under normal process operations do not discharge to the stack. They are used to vent process gas to the stack in the event of a process upset and are not considered a flow contributor or a disturbance.

Per EPA Method 1, a total of 12 traverse points (6 per axis) will be used for M0010 isokinetic sampling. Figure 3-1 provides a schematic of the test ports and traverse point locations.

3.2 VE SOUTH SCRUBBER STACK

Two 6" ID test ports will be installed on the \sim 42" ID steel stack. The ports will be placed \geq 84" (2 diameters) from the location where the waste gas scrubber vent enters the stack. This will allow access to the ports from the roof by scaffold. The ports will be \sim 35' (10 diameters) from the stack exit.

Per EPA Method 1, a total of 24 traverse points (12 per axis) will be used for M0010 isokinetic sampling. It should be noted that near the proposed port locations are a number of small ducts leading to the stack. As is the case with the Division stack, these are catch pots which, under normal operation, do not discharge to the stack. They are used to vent process gas to the stack in the event of a process upset. For the purpose of test port location, and given the fact that there is no flow from these catch pots, they are not considered a flow contributor or a disturbance.

See Figure 3-2 for a schematic of the test port and traverse point locations.

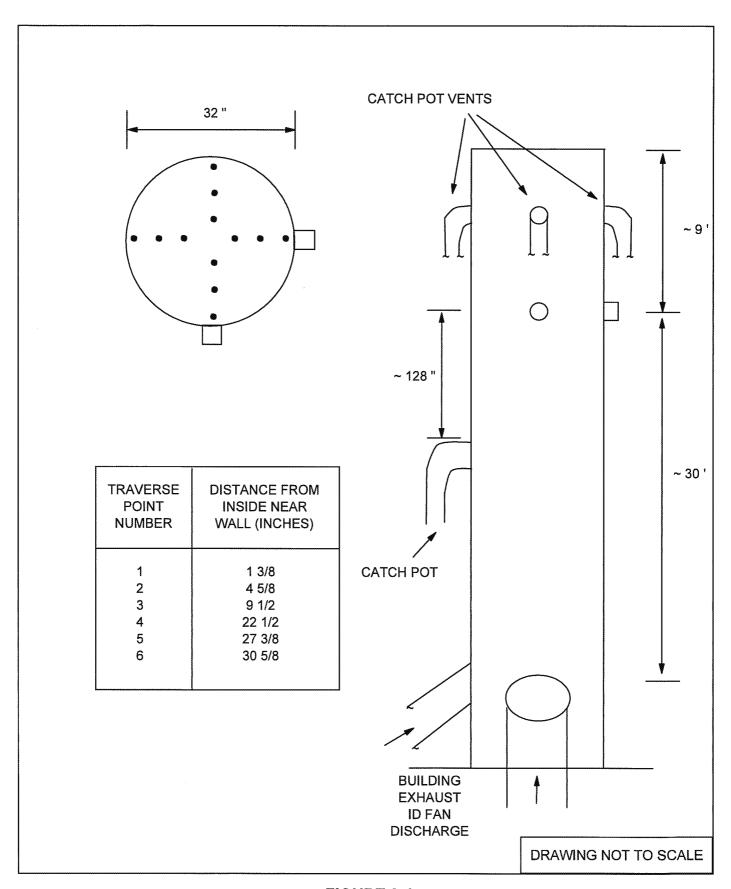


FIGURE 3-1
DIVISION STACK TEST PORT
AND TRAVERSE POINT LOCATION

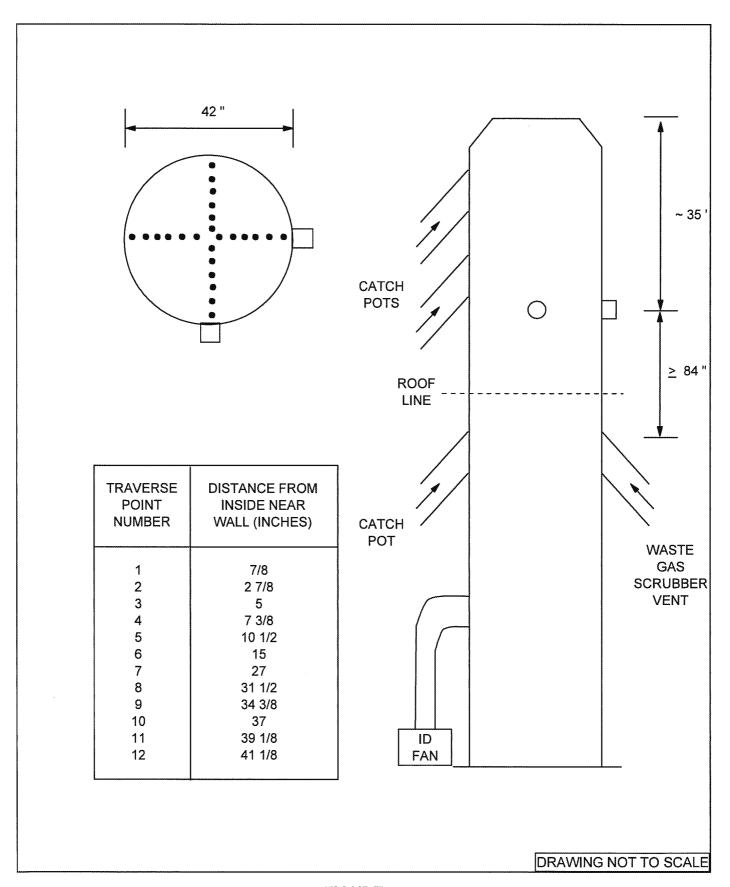


FIGURE 3-2
VE SOUTH SCRUBBER STACK TEST PORT
AND TRAVERSE POINT LOCATION

3.3 PPA PROCESS STACK

Two 4" ID test ports are in place on the ~ 28 " ID fiberglass stack. The ports are 18' (7.7 diameters) from the nearest downstream disturbance (waste gas scrubber demister duct) and 50' (21.4 diameters) from the nearest upstream disturbance (stack exit.)

Per EPA Method 1, a total of 12 traverse points (6 per axis) will be used for M0010 isokinetic sampling. See Figure 3-3 for a schematic of the test port and traverse point locations.

Note: All measurements at all three test locations will be confirmed prior to sampling.

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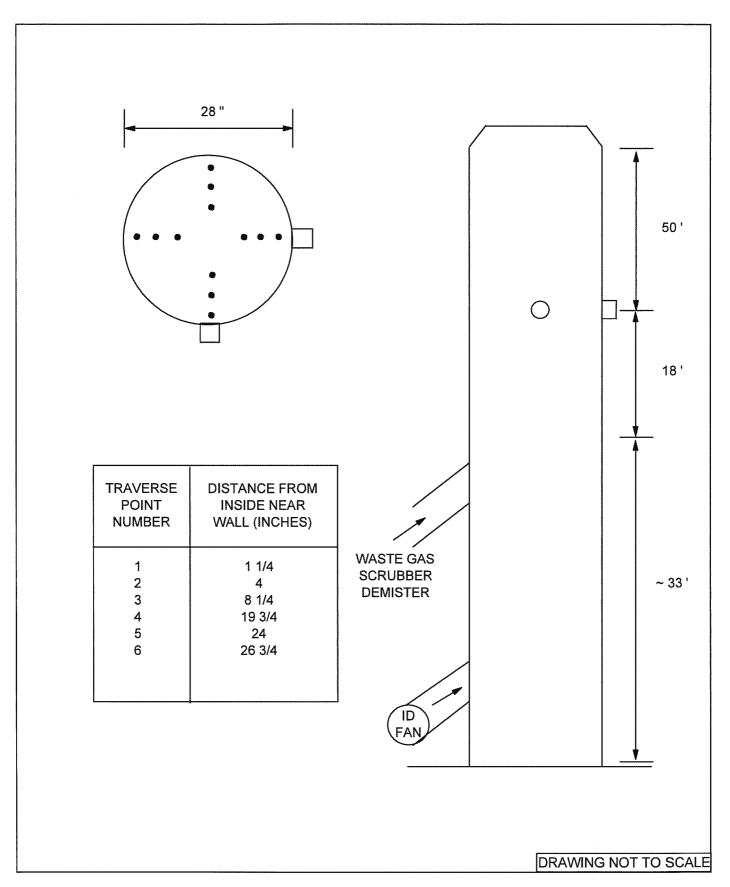


FIGURE 3-3
PPA EXHAUST STACK TEST PORT
AND TRAVERSE POINT LOCATION

4. SAMPLING AND ANALYTICAL METHODS

4.1 STACK GAS SAMPLING PROCEDURES

The purpose of this section is to describe the emissions sampling trains and to detail the stack sampling and analytical procedures utilized during the emissions test program.

4.1.1 Pre-Test Determinations

Preliminary test data will be obtained at each test location. Stack geometry measurements will be measured and recorded, and traverse point distances verified. A preliminary velocity traverse will be performed utilizing a calibrated "S" type pitot tube and a Dwyer inclined manometer to determine velocity profiles. Flue gas temperatures will be observed with a calibrated direct readout panel meter equipped with a chromel-alumel thermocouple. Preliminary water vapor content will be measured by performance of an EPA Method 4 moisture test or by wet bulb/dry bulb temperature measurements.

A check for the presence or absence of cyclonic flow will be conducted at each test location.

Preliminary test data will be used for nozzle sizing and sampling rate determinations for isokinetic sampling procedures.

Calibration of probe nozzles, pitot tubes, metering systems, and temperature measurement devices will be as specified in Section 5 of EPA Method 5 test procedures.

4.2 STACK PARAMETERS

4.2.1 EPA Method 0010

The test train utilized to perform the HFPO Dimer Acid sampling will be an EPA Method 0010 train (see Figure 4-1). The Method 0010 consists of a borosilicate or stainless steel nozzle that will be attached directly to a heated borosilicate probe. Note to minimize thermal degradation of the HFPO Dimer Acid, the probe and filter will be heated above stack temperature to minimize water vapor condensation before the filter. The probe will be connected directly to a heated borosilicate filter holder containing a solvent extracted glass fiber filter.

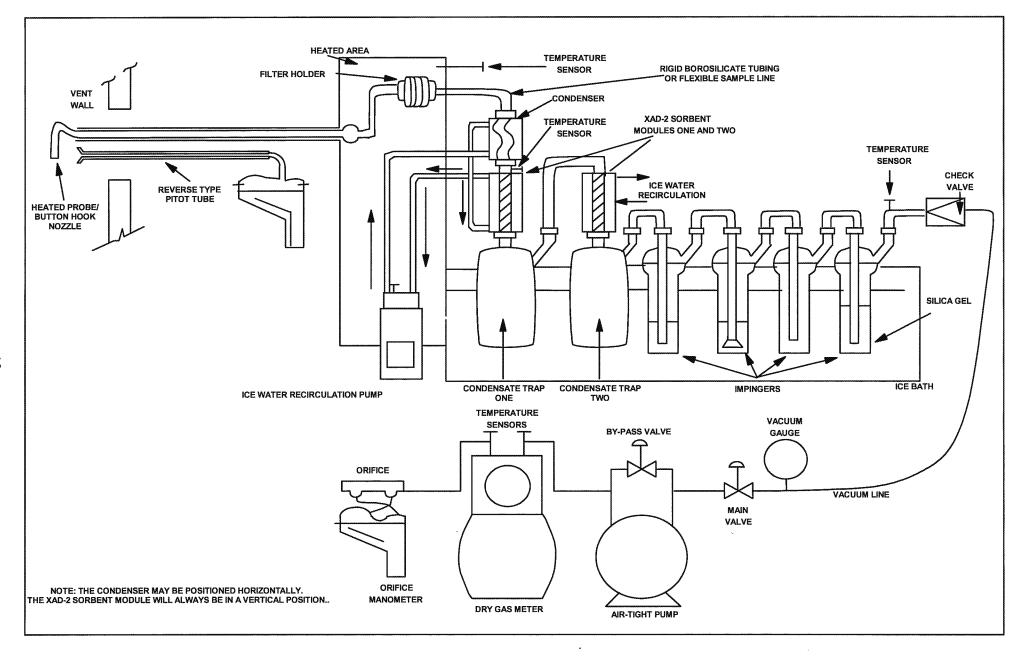


FIGURE 4-1 EPA METHOD 0010 SAMPLING TRAIN

IASDATA\CHEMOURS\15418.002.001\FIGURE 4-1 METHOD 0010-2

A section of borosilicate glass or flexible polyethylene or teflon tubing will connect the filter holder exit to a Grahm (spiral) type ice water-cooled condenser, an icewater-jacketed sorbent module containing approximately 40 grams of XAD-2 resin, and an XAD inlet temperature sensor. The train will also include a second XAD-2 resin trap to minimize any additional condensed water through the trap and to evaluate sample train collection efficiency. Each resin trap will be connected to a 1-L condensate trap. The second XAD-2 resin trap will be followed by a series of three impingers. The first two impingers will each contain 100-ml of high purity distilled water. The final impinger will contain 300 grams of dry pre-weighed silica gel. All impingers and the condensate traps will be maintained in an ice bath. Ice water will be continuously circulated in the condenser and both XAD-2 modules to maintain method required temperature. A control console with a leakless vacuum pump, a calibrated orifice, and dual inclined manometers will be connected to the final impinger via an umbilical cord to complete the sample train.

During sampling, gas stream velocities will be measured by attaching a calibrated "S"-type pitot tube into the gas stream adjacent to the sampling nozzle. The velocity pressure differential will be observed immediately after positioning the nozzle at each traverse point, and the sampling rate adjusted to maintain isokineticity ± 10. Flue gas temperature will be monitored at each point with a calibrated panel meter and thermocouple. Isokinetic test data will be recorded at each traverse point during all test periods, as appropriate. Leak checks will be performed on the sampling apparatus according to reference method instructions, prior to and following each run, component change (if required) or during midpoint port changes.

4.2.2 EPA Method 0010 Sample Recovery

At the conclusion of each test, the sampling train will be dismantled, the openings sealed, and the components transported to the field laboratory trailer for recovery.

A consistent procedure will be employed for sample recovery:

1. The two XAD-2 covered (to minimize light degradation) sorbent modules (1 and 2) will be sealed and labeled.

- 2. The glass fiber filter(s) will be removed from the holder with tweezers and placed in a polyethylene container along with any loose particulate and filter fragments.
- 3. The particulate adhering to the internal surfaces of the nozzle, probe and front half of the filter holder will be rinsed with methanol into a polyethylene container while brushing a minimum of three times until no visible particulate remains. Particulate adhering to the brush will be rinsed with methanol into the same container. The container will be sealed.
- 4. The volume of liquid collected in the first condensate trap will be measured, the value recorded, and the contents poured into a polyethylene container. The sample container will be sealed.
- 5. All train components between the filter exit and the first condensate trap will be rinsed with methanol. The solvent rinse will be placed in a separate polyethylene container and sealed.
- 6. The volume of liquid in the second condensate trap and impingers one, two, and three will be measured, the values recorded, and sample will be placed in a polyethylene container and sealed.
- 7. The second condensate trap, the three impingers and connectors will be rinsed with methanol. The solvent sample will be placed in a separate polyethylene container and sealed.
- 8. The silica gel in the final impinger will be weighed and the weight gain value recorded.
- 9. Site (reagent) blank samples of the methanol, XAD resin, filter and distilled water will be retained for analysis.

Each container will be labeled to clearly identify its contents. The height of the fluid level will be marked on the container of each liquid sample to provide a reference point for a leakage check during transport. All samples will be maintained cool.

During each test run, a M0010 blank train will be setup near the test location, leak checked (twice) and recovered along with the respective sample train. Following sample recovery, all samples will be transported to the TestAmerica Inc. for sample extraction and analysis.

See Figure 4-2 for a schematic of the M0010 sample recovery process.

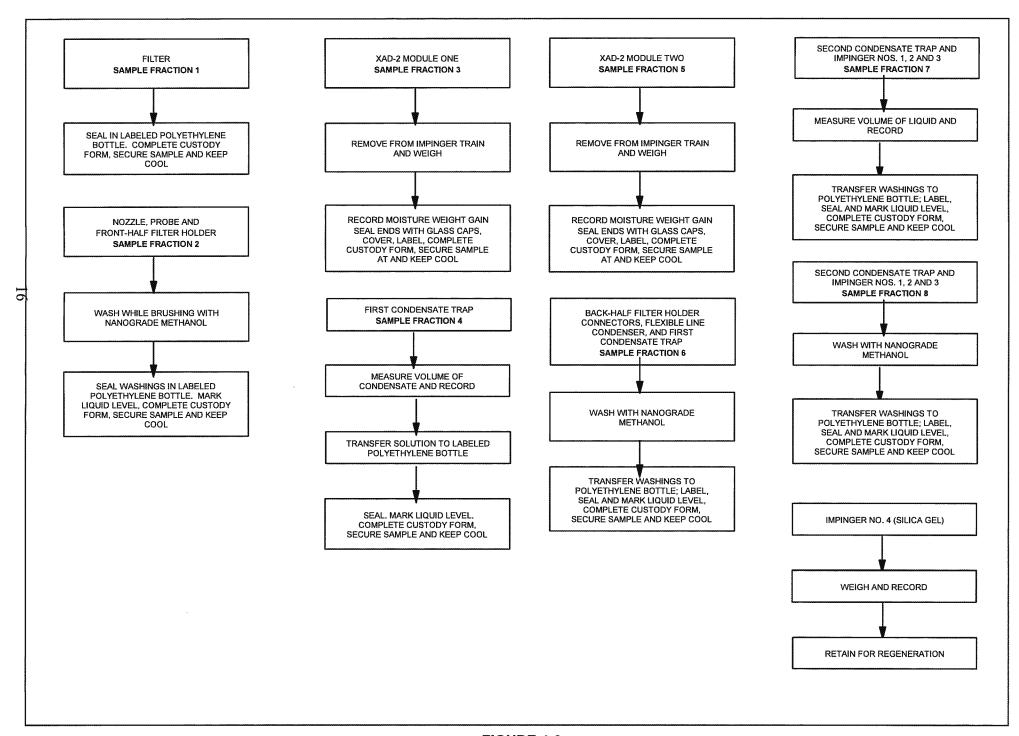


FIGURE 4-2
HFPO DIMER ACID SAMPLE RECOVERY PROCEDURES FOR METHOD 0010

IASDATA\CHEMOURS\15418.002\FIGURE 4-2 EPA 0010-2

4.2.3 EPA Method 0010 - Sample Analysis

Method 0010 sampling trains will result in three separate analytical fractions for HFPO Dimer Acid analysis according to SW-846 Method 3542:

- Front-Half Composite—comprised of the Particulate Filter, and the probe, nozzle, and front-half of the filter holder solvent rinses,
- Back-half Composite—comprised of the XAD-2 one resin material and the back-half of the filter holder with connecting glassware solvent rinses,
- Condensate Composite—comprised of the aqueous condensate and the contents of Impingers #1, 2, and 3 with solvent rinses.

The second XAD-2 resin material will be analyzed separately to evaluate breakthrough.

The Front and Back-half composites and the second XAD-2 resin material will be placed in polypropylene wide-mouth bottles and tumbled with methanol for 24 hours. Portions of the extracts will be processed analytically for the HFPO dimer acid by Liquid Chromatography and duel mass spectroscopy (HPLC/MS/MS). The Condensate composite will be concentrated onto a solid phase extraction (SPE) cartridge followed by desorption from the cartridge using methanol. Portions of the final extracts will also be processed analytically by HPLC/MS/MS.

Samples will be spiked with isotope dilution internal standards at the commencement of their preparation to provide accurate assessments of the analytical recoveries. Final data will be corrected for Isotope Dilution standard recoveries.

Test America Inc. is in the process of development of procedures for the sample extraction and analysis for HFPO Dimer Acid. Once these procedures are available, they will be incorporated into this Test Protocol.

4.3 GAS COMPOSITION

A mobile laboratory equipped with instrumental analyzers will be used to measure carbon dioxide (CO₂), and oxygen (O₂) concentrations. A diagram of the WESTON sampling system is presented in Figure 4-3.

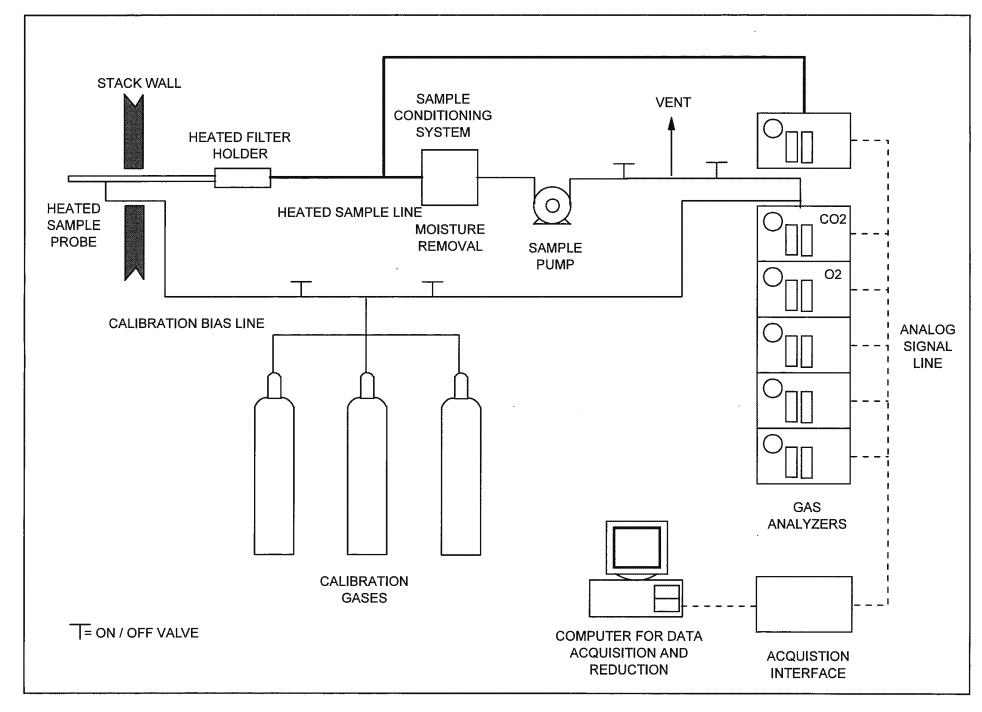


FIGURE 4-3
WESTON SAMPLING SYSTEM

IASDATA\CHEMOURS\15418.002.001\FIGURE 4-3 WESTON SAMPLING SYSTEM

A heated stainless steel probe will be used to collect a sample at the approximate centroid of each stack. At the end of the probe, a tee will permit the introduction of calibration gas. The sample will be drawn through a heated Teflon® sample line to the sample conditioner. The output from the sampling system will be recorded electronically, and one-minute averages will be recorded and displayed on a data logger. As an option, the sample may be collected at the exhaust of the Method 0010 sampling system.

Each analyzer will be set up and calibrated internally by introduction of calibration gas standards directly to the analyzer from a calibration manifold. The calibration manifold is designed with an atmospheric vent to release excess calibration gas and maintains the calibration at ambient pressure. The direct calibration sequence will consist of alternate injections of zero and midrange gases with appropriate adjustments until the desired response is obtained. The high range standards will then be introduced in sequence without further adjustment.

The sample line integrity will be verified by performing a bias test before and after each test period. The sampling system bias test consists of introducing the zero gas and one up range calibration standard in excess to the valve at the probe end when the system is sampling normally. The excess calibration gas flows out through the probe to maintain ambient sampling system pressure. Calibration gas supply is regulated to maintain constant sampling rate and pressure. Instrument bias check response will be compared to internal calibration responses to insure sample line integrity and to calculate a bias correction factor after each run using the ratio of the measured concentration of the bias gas certified by the calibration gas supplier.

The oxygen and carbon dioxide content of each stack gas will be measured according to EPA Method 3A procedures which incorporate the latest updates of EPA Method 7E. A Servomex Model 4900 analyzer (or equivalent) will be used to measure oxygen content. A Servomex Model 4900 analyzer (or equivalent) will be used to measure carbon dioxide content of the stack gas. Both analyzers will be calibrated with EPA Protocol gases prior to the start of the test program and performance will be verified by sample bias checks before and after each test run.

5. TEST PROCEDURES

Each test will be a minimum of 180 minutes in duration and sufficient to collect $\geq 3 \text{m}^3$ (105.9 ft³) of sample volume. A total of three test runs per location will be performed.

6. TEST REPORT FORMAT

A written final report will be prepared for NC DEQ. The report will be submitted within 90 days of test completion. The test report will include:

- 1. A summary section which will contain a concise description of the Emissions Test program including reasons for testing, number and types of tests, technical approach, etc. A summary table showing key test data and results.
- 2. A description of the test locations and sampling equipment used for the tests.
- 3. A description or reference of sampling and analytical procedures.
- 4. Test data and results summary tables. Emissions will be reported on a concentration basis (lb/dscf, ug/m³) and mass basis (lb/hr, gm/sec).
- 5. A discussion section will include comments concerning any unusual process conditions or difficulties experienced with testing or analytical procedures.
- 6. Appendices containing:
 - Process operations data (process operating and control data to be monitored and supplied by Chemours).
 - Raw and reduced test data.
 - Laboratory analytical report.
 - Sample calculations.
 - Equipment calibration records.
 - List of project participants.

7. QUALITY ASSURANCE AND CALIBRATIONS

The following procedures will be followed to insure that the emissions data collected during this test program are of high quality.

7.1 VELOCITY/VOLUMETRIC FLOW RATE QC PROCEDURES

Volumetric flow rates will be determined using EPA Methods 1 and 2. The following QC steps will be followed during these tests:

- The pitot tube will be visually inspected before sampling.
- Both legs of the pitot tube will be leak-checked before sampling.
- The manometer oil will be leveled and zeroed before each run and periodically checked.
- Cyclonic flow checks using an S-type pitot tube will be performed once per source during testing.
- Pitot tube coefficients will be determined based on physical measurement techniques as delineated in Method 2.

7.2 MOISTURE CONTENT AND SAMPLE VOLUME QC PROCEDURES

Gas stream moisture will be determined by EPA Method 4 as part of the M0010 isokinetic stack tests. The following QC procedures will be followed in determining the volume of moisture collected:

- The amount of moisture collected will be determined either volumetrically or gravimetrically.
- The balance will be calibrated daily.
- The balance zero will be checked and re-zeroed if necessary before each weighing.
- The balance will be leveled and placed in a clean, motionless environment for weighings.
- The indicating silica gel will be fresh for each run and periodically inspected and replaced during runs if needed.

The QC procedures that are followed in regard to accurate sample gas volume determination will be:

- The dry gas meter will be fully calibrated annually using an EPA-approved intermediate standard device.
- Pre-test, port-change, and post-test leak-checks will be completed (must be less than 0.02 cubic feet per minute (cfm) or 4% of the average sample rate).
- The gas meter will be read to the thousandth of a cubic foot for all initial and final readings.
- Readings of the dry gas meter, meter orifice pressure (Delta H) and meter temperatures will be taken at every sampling point.
- Accurate barometric pressures will be recorded at least once per day.
- Pre- and post-test program dry gas meter checks will be completed to verify the accuracy of the meter calibration constant (Y).

7.3 METHOD 0010 SAMPLING QC PROCEDURES

The specific sampling QC procedures which will ensure representative collection of Method 0010 data are as follows:

- Pre-clean sample and recovery glassware as described in Method 0010.
- Prescreen each lot of XAD resin prior to packing the XAD resin trap in preparation for sampling. All screening and packing will be performed at the laboratory prior to shipment to the field.
- Only nanograde or equivalent solvents will be used for test train preparation and recovery. High purity distilled water will be used.
- The sample rate must be within 10% of the true isokinetic (100%) rate.
- All sampling nozzles will be manufactured and calibrated according to EPA standards.
- Maintain the XAD-2 resin gas stream inlet and silica gel exit gas stream temperature at or below 68 °F during testing.
- Complete recovery procedures in a clean environment.

- Maintain solvent, XAD and filter samples cool following collection and prior to analysis.
- All calculations will be double-checked for accuracy and completeness.
- All manual test apparatus will be leak checked before and after each test run. Each leak check will be recorded on a field sampling data sheet.

7.4 INSTRUMENTAL ANALYZER QC SAMPLING PROCEDURES

The following QC procedures will be applied to ensure collection of representative test data.

- The gas composition sampling systems (probe to sample conditioner) will be leak-checked prior to the testing.
- All analyzers will be calibrated prior to testing to ensure precise and accurate data. Protocol standards will be used to calibrate each of the analyzers. Each analyzer will be calibrated at three to four points (zero, mid, and high range) depending on reference method requirements. Nitrogen will be used to set the instrument zero. The calibration standards will be approximately 40-60 and 100% span for CO₂ and O₂.
- Prior to formal testing, a stratification check will be performed on each stack. Per Section 8.1.2 of EPA Method 7E, a three point CO₂ or O₂ stratification check passing through the centroidal area of the stack will be performed. The three points (16.7, 50 and 83.3) percent of the stack diameter will each be sampled a minimum of two times the system response. If the CO₂ or O₂ concentration at each point in comparison to the mean difference is no more than ± 5.0 percent, then all sampling will be performed at a single point at the stack midpoint. If the difference is between 5 percent but not greater than 10 percent, a three point traverse per run will be performed. A difference of greater than 10 percent in concentration will result in performing a twelve point traverse with points calculated per EPA Method 1.
- Pre- and post-test calibration bias tests will be performed for each test run. The bias check will be performed with the calibration standard that is closest to the observed concentration in the sample gas. The average pre/post-test bias drift will not exceed 3 % of full scale.
- A permanent data record of analyzer responses will be recorded using computer software.
- If preliminary measurements of O₂ and CO₂ indicate that the stack gas concentrations are equivalent to ambient air (20.9% O₂, 0% CO₂), then further O₂ and CO₂ monitoring may not be required.

7.5 DECONTAMINATION PROCEDURES

Stack gas sampling equipment will be pre-cleaned following standard source test method procedures. All stack gas sampling equipment will be cleaned on-site as part of individual sample recovery procedures.

Sample containers are purchased from a vendor. The vendor provides a certificate indicating that each lot of bottles is free of contaminants.

All personnel associated with sample collection will use designated personal protective equipment (PPE). Personnel will follow standard PPE decontamination procedures for each level of PPE required.

Personnel have received the proper hazardous materials training specified in 29 CFR 1910.